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(54) Title: **METHOD AND APPARATUS FOR FORMING A FILM ON A SUBSTRATE**

(57) Abstract: This invention relates to a method and apparatus for forming a film on the substrate. The method comprises supplying to the chamber in gaseous or vapour form a silicon containing organic compound and an oxidising agent in the presence of a plasma to deposit a film on the substrate and setting the film such that carbon containing groups are retained therein. In particular embodiments the setting is achieved by exposing the film to H₂ plasma.

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Method and Apparatus for forming a Film on a Substrate

This invention relates to a method and apparatus for forming a film on a substrate particularly, although not exclusively, to a film which is deposited on a semiconductor silicon wafer with flowing properties and retains carbon-containing groups on setting.

A number of methods have been disclosed for depositing a thin film on a semiconductor wafer, and examples included US 5314724, US 489753, US 5593741, EP-A-0731982 and EP-A-0726599. It can be seen from these that, up until recently, organic-containing silicon precursors have been processed or further processed in such a way as to avoid or subsequently remove organic components from the as-deposited film; this is, for example, disclosed in US 5314724. In addition, for some applications, it has been found to be difficult to retain both good film quality and good gap-fill capability on the surfaces and in recesses of a wafer on which the film is deposited.

According to a first aspect of the present invention, there is provided a method of forming a film on a substrate comprising:

- (a) positioning the substrate on a support in a chamber;
- (b) Supplying to the chamber in gaseous or vapour form a silicon-containing organic compound and an oxidising agent in the presence of a plasma to deposit a film on the substrate; and
- (c) setting the film such that carbon-containing groups

are retained therein.

The present invention provides a method of forming an unset film. Typically, the film is flowable (that is a film with a degree of surface mobility) and may thus provide good gap fill properties on the substrate.

It should be understood that without affecting the generality of this invention where gap fill properties are not required, e.g. for InterMetal Dielectric (IMD) layers on a semiconductor, then a flowing intermediate film will not be required. However the intermediate film deposited according to this invention will contain OH, which is almost entirely removed by the 'setting' process.

The formation of an OH and CH containing intermediate which is then further processed to remove OH but contain CH enables the formation of improved dielectric layers.

The substrate may be a semiconductor wafer, for example a silicon semiconductor wafer of the type known in the art.

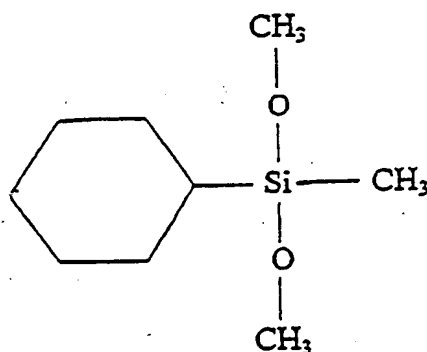
Preferably, the oxidising agent is oxygen, although others could be used, for example H_2O_2 .

For example, the silicon-containing organic compound may be an organosilane or an organosiloxane. Preferably, the silicon-containing organic compound is an alkylsilane, and even more preferably is a tetraalkylsilane. In a particularly preferred embodiment of the invention, the silicon-containing organic compound is tetramethylsilane (TMS). However, for example, other organosilanes or

organosiloxanes could be used, one example being 1,1,3,3-tetramethyldisiloxane (TMDS).

Experiments suggest that methoxysilanes, and in particular methoxymethylsilanes, produce films with very low dielectric constants and may be particularly preferred.

Particularly good results have been achieved with cyclohexyldimethoxymethylsilane (CHDMMS) which has the following structure:



Experiments have also shown that a methoxysilane (e.g. CHDMMS) may be able to be processed as in the above method described, but without any oxidising agent present in the plasma. It is supposed that this is because the Si-O bond already exists as part of the methoxy group.

Accordingly, according to another aspect the invention consists in a method of forming a film on a substrate comprising:

- (a) positioning the substrate on a support in a chamber;
- (b) supplying to the chamber in gaseous or vapour form an organic compound including an Si-O bond to deposit a

film on the substrate; and

(c) setting (e.g. annealing) the film such that carbon-containing groups are retained therein.

5 Preferably the compound is supplied in the presence of a plasma, but other energy sources may be utilised to cause appropriate deposition and these may be combined with spin-on techniques.

10 The film may be deposited on a substrate positioned on a low-temperature support for example, a support at a temperature of about 0°C. Indeed temperatures in the range 0°C to 70°C have produced practical results, with temperatures of 30° and 50°C proving particularly practical.

15 In one embodiment, the method may further comprise supplying RF power during deposition of the film. This RF power is preferably applied to a showerhead or the like through which the gaseous precursors are passed into the chamber.

20 Whilst any suitable experimental conditions may be used, it has been found that typical conditions include a flow rate of 210 sccm tetramethylsilane, a flow rate of 200 sccm O₂, a chamber pressure of 2000 mT, a support temperature of 0° and a showerhead temperature of 100°C, and 250 watts of 380 khz RF power applied to the
25 showerhead, although it is pointed out that these are only typical conditions.

The setting of the film may be carried out by an

annealing step, for example at a temperature of about 450°C which serves to remove water from the deposited film. It has been found that typical k value of the set film is about 2.55, for example for a 6000Å thick film deposited with a base layer (prior to deposition of the film) or capping layer (on the formed layer) after an annealing step at about 450°C in the absence of oxygen. This k value is a measure of the dielectric constant and it can be seen that the present invention provides a particularly low dielectric constant.

Alternatively, and often preferably, the setting step is performed by exposing the deposited film to an H₂ containing plasma without any prior heating of the film. In this connection it is preferred that the support is not biased during the deposition stage to avoid heating arising from ion bombardment.

According to a third aspect of the present invention, there is provided a method of forming a film on a substrate comprising:

- (a) positioning the substrate on a support in a chamber;
- (b) supplying to the chamber in gaseous or vapour form tetramethylsilane and oxygen in the presence of a plasma and a supply of RF power to deposit a film on the substrate; and
- (c) setting the film such that carbon-containing groups are retained therein.

The setting step can be performed as set out above.

Particularly good results have been achieved with the H₂ plasma treatment without prior heating.

According to a further aspect of the present invention, there is provided an apparatus for forming a film on a substrate, the apparatus comprising:

- (a) a support for the substrate positioned in a chamber;
- (b) means for supplying to the chamber in gaseous or vapour form a silicon-containing organic compound and an oxidising agent in the presence of a plasma to deposit a film on the substrate; and
- (c) means for setting the film such that carbon-containing groups are retained therein.

The apparatus may, in one embodiment, further comprise means for improving the uniformity of the deposition of the film on the substrate. This may be arranged in the region of, or around, a showerhead and, whilst the applicant is not to be restricted hereby, it is thought that its role in the uniformity of deposition is possibly as a result of providing a site for surface reactions about the surface periphery thus enhancing deposition rate at the edge of the substrate.

Although the invention has been defined above, it is to be understood that it includes any inventive combination of the features set out above or in the following description.

The invention may be performed in various ways and a specific embodiment will now be described, by way of

example, with reference to the accompanying drawings, an in which:

Figure 1 is a schematic view of an apparatus for use in the present invention;

5 Figure 2 is a Fourier Transform Infra-Red (FTIR) spectrum showing an as-deposited and annealed film according to this invention;

10 Figures 3(a) and 3(b) are scanning electron micrographs showing the annealed film formed by the present invention;

Figure 4 is a Fourier Transform Infra-Red (FTIR) spectrum for a first process run without oxygen;

Figure 5 is the equivalent FTIR for the process run with oxygen;

15 Figure 6 is a table showing initial experimental results using standard delivery systems for CHDMMS;

Figure 7 is a table showing experimental results using a syringe pump to deliver CHDMMS;

20 Figures 8 to 10 are FTIR spectrum relating to certain experiments identified in Figure 7;

Figure 11 shows FTIR plots for film formed from TMS after an anneal (FTM) process and after H₂ plasma processing when no anneal step has occurred;

25 Figure 12 is a graph of plots showing the affect of an FTM process, a 5 min H₂ plasma and a 10 min H₂ plasma on the thickness and the refractive index of a TMS based film;

Figures 13 to 15 are respective bar graphs indicating

the affects of an oxidising strip on FTM, 5 min H₂ plasma and 10 min H₂ plasma treated TMS + O₂ deposited films;

Figure 16 illustrates the film stress values pre- and post-oxidising strip, after various post film formation treatment regimes;

Figure 17 sets out the dielectric constant of TMS films after various post formation regimes;

Figure 18 is a bar graph comparing the dielectric constant of layers treated by FTM, 5 min, 10 min and 30 min H₂ plasma for a pair of substrates; and

Figure 19 is a SIMS plot for a TMS + O₂ deposited film which has been set with a 5 min H₂ plasma.

Referring to Figure 1, there is shown an apparatus generally at 1 which includes a vacuum chamber 2 having a showerhead 3 and a wafer support or platen 4. The showerhead 3 is connected to an RF source (not shown) to form one electrode, whilst the support 4 if preferably earthed. Alternatively or additionally, the RF source could be connected to the support 4. The showerhead 3 is connected via pipes (not shown) to respective sources of tetramethylsilane and oxygen. The apparatus is generally of the form disclosed in EP-A-0731982, which is incorporated herein by reference. However, a standard (non-duplex) showerhead is normally used. Also shown is an optional uniformity ring 5 arranged around the showerhead 3. This ring 5 plays an active role in the uniformity of deposition of the film on the wafer and may be desirable

for some processes.

In use, the apparatus 1 is arranged to deposit a water and/or OH containing intermediate layer on a wafer which may be flowable and may be used to produce a planar layer or for "gap filling" for applications such as pre-metal dielectric, shallow trench isolation and intermetal dielectric on semiconductor devices. The film is formed by introducing into the chamber tetramethylsilane and oxygen in gaseous or vapour form and reacting them within the chamber. This forms an intermediate layer with a degree of surface mobility even when a plasma is present. It has been found that very small dimensioned gaps can be filled by the process of the present invention. This is not to restrict the invention as often process conditions may yield an intermediate layer containing OH that does not flow but still retains the other characteristics of this invention. The reaction takes place in the presence of a plasma. Subsequently, the film is annealed by heating, preferably in the absence of oxygen and most preferably in the presence of a hydrogen containing plasma.

Example

The precursor tetramethylsilane (TMS) has been applied with various other precursors, a platen temperature of approximately 0°C and RF power of either low (e.g. 380 Khz) or high (e.g. 13.56 Mhz) frequency where indicated. The basic results were as follows:

	TMS + H ₂ O ₂	No deposition in the pressure range up to 5,000 mT
5	TMS + MeSiH ₃ + H ₂ O ₂	Slight increase in carbon content over just MeSiH ₃ + H ₂ O ₂ . Deposition rate ~ 6000 Å/min
	TMS + DiH ₄ + H ₂ O ₂	No carbon in the film. Deposition rate ~ 900 Å/min
	TMS + H ₂ O ₂ + RF	Deposition rate ~ 400 Å/min. High refractive index
10	TMS + O ₂ + RF	Deposition rate over 2 micron/min - high carbon content.

A 'preferred' process was then developed consisting of:

210sccm TMS (calculated from fill rate checks)

200sccm O₂

15 2,000 mT pressure

0° platen temperature and 100°C showerhead temperature

250 watts of 380khz RF power applied to the showerhead.

This yielded a k value of 2.55 (measured by CV techniques)

20 for a 6,000 Å thick film deposited without base or capping after an anneal at approximately 450°C in the absence of oxygen.

Figure 2 shows a FTIR spectrum for an as-deposited and annealed film according to this aspect of the invention. The two spectra are shown overlain on the same diagram for ease of comparison. The as-deposited spectrum is the lower of the two and shows at 6 the characteristic peak of O-H bonds associated with water. Between 3,000 and 2,600 wave

numbers O-H bonds associated with free water, isolated O-H and H bonded O-H are present. Water contains free water and H bonded O-H and thus gives a characteristically broad peak in this area. At 7 is the C-H₃ peak; at 8 is the Si-CH₃ peak (Si-C); and at 9 is the Si-O peak.

It can thus be seen that a film has been deposited containing water and/or OH which is subsequently removed by the annealing step and that CH₃ is present, is bonded to Si and remains in the film after annealing to form the hard film.

Generally, an indicator of low k characteristics is a high Peak Area Ratio(PAR) between Si-C and Si-O on the FTIR. It is believed Si-C bonds block Si-O bonds and thus reduce the density of the resultant film. Hence, a high Peak Area Ratio Si-C:Si-O is indicative of a low k film. It was however noted that for these plasma deposited and annealed films the measured k values were not as low as the Peak Area Ratio Si-C:SiO would suggest from non-plasma deposited low k films deposited from a reaction of methylsilane and peroxide.

Films of this invention as annealed as shown in Figure 3 which demonstrate the flowing characteristics of the as-deposited film.

In general, the following effect of changing the parameters in a process have been observed:

	<u>Parameter</u>	<u>Property</u>			
		<u>Refractive Index</u>	<u>FTIR Peak SiC/SiO</u>	<u>Area Ratio CH/SiO</u>	<u>Uniformity</u>
5	Pressure increase	down	up	up	better
	Power increase	none	down	down	better
	Nitrogen flow increase	up	down	down	worse
	TMS/O ₂ ratio increase	down	up	up	none
10	Total TMS/O ₂ decrease	none	down	down	none

An experiment has particularly been carried out using cyclohexyldimethoxymethylsilane (CHDMMS). As is reported below this has shown significantly reduced dielectric constants. It is anticipated that benefits will be found from many methoxysilane compounds such as tetramethoxysilane.

The experiments were carried out in a chamber substantially as shown in Figure 1, or in our co-pending British Patent Application 9914879.3, with an electrode gap spacings of 40mm and 20mm and the uniformity ring shield used for non-plasma based processes removed. The CHDMMS was fed into the chamber using a syringe delivery system described in our co-pending British Patent Application No. 9922691.2, which is incorporated hereinby reference, as opposed to a traditional low vapour pressure mass flow controller. This was done due to the fact that, as described below, CHDMMS could not be reliably delivered by conventional means as it has a relatively high boiling point (approximately 200°C) compared to most of the other precursor materials investigated in Application 9914879.3.

All processes were run with plasmas applied to the

showerhead. All wafers were 'set' by annealing for typically 30 minutes at approximately 480°C.

The following parameter ranges have been investigated:

5	Pressure	-	500 mT to 1500 mT
	Power (380 kHz)	-	50 W to 750 W
	Platen temperature	-	0°C to 70°C
	CHDMMS flows	-	0.5 g/min to 1.5 g/min
	Oxygen flows	-	0 to 200 sccm
10	Nitrogen flows	-	0 to 400 sccm
	Peroxide flows	-	0 to 0.75 g/min

It will be appreciated that the relative flow rates are particularly relevant to the process. In general higher rates lead to higher deposition rates and thus a broad range of flow rates can achieve similar results. Thus values outside the above ranges may be applicable.

Two particularly preferred process examples are given below: one of these is with oxygen and one is without oxygen.

Process 1 (no O₂)	
Pressure	900 mT
Power	500 W
Platen temperature	50°C
CHDMMS flow	0.85 g/min
Nitrogen flow	200 sccm

Process 2 (with O₂)	
Pressure	900 mT
Power	250 W
Platen temperature	50°C
CHDMMS flow	0.85 g/min
Oxygen flow	50 sccm
Nitrogen flow	150 sccm

The resultant films were annealed and the post anneal results were as follows:

Process 1 (no O₂)	
Deposition rate	17000Å/min
Uniformity (max/min)	± 4%
Refractive index	1.370
Dielectric constant	2.55

Process 2 (with O₂)	
Deposition rate	9500Å/min
Uniformity (max/min)	± 5%
Refractive index	1.340
Dielectric constant	2.25

5 As can be seen the dielectric constants in each case are desirably low, but the "with oxygen" process is significantly advantageous.

10 Figures 4 and 5 show the respective FTIR spectra post anneal. It will be seen that they are substantially similar. The feature between 2500 and 2000 in Figure 5 is believed to result from atmospheric (background) CO₂.

15 In fact, initial experiments were carried out using a CHDMMS source consisting of a PTFE pot within an evacuated aluminium vessel which was heated to 150°C. The pot was connected by gas line to a gas mass flow controller suitable for H₂O with a conversion factor of 1.000. The RF power was applied to the showerhead with a spacing from the wafer of 40mm. The RF was 380khz continuous mode. Results from these experiments are shown in Figure 6. The numbers

in the CHDMMS column are the nominal gas flow as measured by the mass flow controller however stable flows could not be achieved and therefore these results are for near random quantities of CHDMMS being delivered to the process chamber. At this point experimentation was halted until a superior delivery system for this precursor could be developed.

CHDMMS has a boiling point of 201.2°C, and a density of 0.940 g/cc. As it was noted in these experiments that CHDMMS deposits a low k insulator without the addition of an oxidising agent it is therefore possible that it could be delivered as a liquid to a semiconductor wafer without a chamber being required (e.g. by well known 'spin-on' techniques) and then reacted either thermally or by plasma to form a low k ($k < 3$) insulator layer. The apparatus used may in effect deposit a liquid by vaporising the liquid precursor, delivering it as a vapour and then condensing it onto the wafer at a temperature below the boiling point of the precursor at that pressure. It is not yet clear if the reactions to the precursor take place on the wafer or at some other place, depositing reaction products onto the wafer.

Having developed a more suitable liquid delivery system which utilises a syringe pump, further experiments

were carried out as shown in Figure 7. From these experiments preferred processes were developed as further described here. FTIR for runs 13, 14 and 16-23 of Figure 6 respectively are illustrated in Figures 8 to 10.

5 Further experiments have been carried out using the following conditions:

Pressure: 2500 millitorr.
 RF Power at 13.56 Mhz 250 watts/200mm wafer
 Showerhead temperature 100°C
 10 TMS flow 100sccm (approximately)
 Oxygen flow 100sccm
 Nitrogen flow 500-600sccm

15 The ratio of TMS (tetramethylsilane) to oxygen is the same as previously (approximately equal quantities), but at half the total flow rates. In this preferred process nitrogen has been used, primarily as a dilutant.

20 Thermal treatment step ("setting" or "anneal")
 Time 5 minutes
 Pressure 10 torr nitrogen (no oxygen)
 Wafer temperature 400°C approximately

25 In the process above, where the process platen temperature was varied, the results were as follows

	T Rate		Non-Uniformity		FTIR		RI av.	Dielectric constant
	°C	Å/min	% max/min	SiC/SiO	SiH/SiO	CH/SiO		
30	10	7,778	2.7	0.0608	0.0060	0.0287	1.3825	2.72
	20	7,673	3.8	0.0594	0.0057	0.0280	1.3832	2.72
	30	7,589	4.8	0.0588	0.0059	0.0282	1.3791	2.65
	45	7,543	3.1	0.0584	0.0056	0.0273	1.3867	2.70
	55	not recorded		0.0527	0.0044	0.0234	not recorded	2.75
35	60	6,968	3.9	0.0512	0.0074	0.0293	1.3935	2.69

40 Once more the initial deposition process puts down a water and/or OH containing intermediate film which needs to be set to substantially remove the water and/or OH to create a low k layer. For the purposes of the above

experiment this setting was achieved by a thermal treatment step as indicated. However other post deposition processes have been experimented with, as can be seen below. The significant features of this experiment were both the continued achievement of a dielectric constant below 3 and the observation that both the refractive index (which is believed to be a measurement of density) and the dielectric constant dipped when a platen temperature of 30° was used. These results are consistent with the normal understanding that dielectric constant and density are related for a specific film composition, so that lower refractive index will normally indicate a lower dielectric constant.

Subsequent to this experiment a further set of films were formed using the following process matrix:

- TMS - 100 sccm
- O2 - 100 sccm
- N2 - 600 sccm
- Pressure - 2000 mT
- Substrate support- 30°C, DC ground potential
(unbiased)
- Power - 250 Watts High Frequency 13.56 MHz
to the showerhead

The films resulting from this process were set either by the annealing process (which is hereinafter referred to

as FTM treatment) or process and/or by treating the film with an H₂ plasma treatment.

The FTM process was as follows:

- Wafer temperature 450°C
- 5 • Pressure 10 Torr (Nitrogen)
- Time 3-5 minutes

The H₂ plasma treatment was as follows:

- Hydrogen - 1000 sccm
- Pressure - 4000 MT
- 10 • Temperature - 400°C
- Power - 2000 Watts High Frequency 13.56 MHz
to an electrode opposed the wafer
- Time - Depends on thickness but typically 3
minutes for 6kÅ although longer times
15 appear to lead to lower K.

Alternatively other RF frequencies could be used applied to any electrode or electrodes either internal or external to the wafer containing chamber such as to create or sustain ionised hydrogen species adjacent to the film to be
20 treated. This is to include remote plasma sources including microwave and inductively completed RF sources wherever situated.

The H₂ plasma may also contain other components e.g. effectively inert dilutants for example argon, helium or

other gasses or vapours that do not detract from this treatment.

Figure 11 shows respective FTIR plots for the film prepared as described above and then treated by FTM or by plasma. Beneath each chart is an indication of the bonding ratios between various components. As will be understood by a man skilled in the art the slope of the graph is not relevant in a FTIR plot; it is only the peaks which provide information. The relative heights of the peaks are indicated by the ratio tables and it will be seen that there is a significant reduction in the bonding between the various components in a plasma treated film as compared with one that has had the FTM post deposition treatment. This indicates that the hydrogen plasma treated intermediate film is structurally different from the FTM treated intermediate film.

In Figure 12 the upper plot indicates that there is no significant difference in film thickness between the various post treatment processes, but there is a significant driving up of the refractive index the longer a plasma treatment takes place. Indeed after a 10 minute H_2 plasma, the refractive index is approaching that of pure SiO_2 . Normally, as has been explained above, this would be expected to result in a significant increase in dielectric

constant, but as can be seen in Figure 17 an increase in the period of the H_2 plasma treatment simultaneously produces a significant decrease in the dielectric constant, provided that the film is not subject to heat treatment prior to the plasma treatment. Thus after a 10 minute H_2 plasma treatment the OH containing film is set and has a dielectric constant of below 2.2, which is an extremely low figure typically unachievable by chemical vapour deposition means.

It will be noted that a very short H_2 plasma treatment (e.g. 1 minute) does not render the film totally stable nor does it reduce significantly the dielectric constant below an FTM anneal although the film is still comparable with other reported films. Wet etch rate experiments have demonstrated that the hydrogen plasma treatment starts at the top of the film. The longer the plasma process, the greater the depth treated the lower the k value of the treated film. The hydrogen plasma treated film etches considerably (e.g. 20 or more times) slower than the FTM treated film. It will also be observed that an H_2 plasma treatment is also not effective in reducing dielectric constant after a previous heating or anneal step.

Turning now to Figures 13, 14 and 15 it will be seen in Figure 13 that film which has had the FMT treatment

appears to be very susceptible to an oxidising process e.g. a photoresist strip with carbon and hydrogen seemingly being removed from the material. This is not totally surprising, because such an oxidising process was previously used to remove such materials in the film (in particular organics from precursors) before it was appreciated that they might have a beneficial affect on the dielectric constant. Figures 14 and 15, on the other hand, show that the plasma treated films are substantially unaffected by the oxidising photoresist strip process. This is of course important, because it makes it much easier to remove resist from the surface of the dielectric layer without damaging that layer. Figure 16 shows that the plasma treated films also have particularly low stress values whether before or after the oxidising strip.

It is believed that similar results would be achieved with most flowable or water and/or OH containing films that retain carbon, more particularly CH, in the finally used dielectric layers.

Thus the inventors have, in particular determined a process for depositing a flowable or at least OH containing intermediate film which is subsequently set using heat in the absence of oxygen and preferably in the presence of an H_2 containing plasma with the result that the film has a

dielectric constant below 3 and, with the application of hydrogen plasma, a reduced dielectric constant and a good resistance to oxidising strip. It is postulated that this combination results from the surprising fact that H₂ treatment reduces the dielectric constant whilst increasing the refractive index and hence, almost certainly the density as evidenced by a greatly reduced wet etch rate.

Further evidence for this is illustrated in Figures 18 and 19. Figure 18 shows that progressive treatment by H₂ plasma drives down the dielectric constant, with samples treated for 30 minutes achieving a k of 1.8.

Figure 19 provides analysis of the hydrogen plasma treated film has been carried out as follows:

SIMS (secondary ion mass spectrometry) data of a TMS + O₂ deposited film that has been hydrogen plasma treated for 5 minutes has been generated. [The horizontal axis is through the depth of the sample starting at just above the surface and ending in the silicon wafer. What is shown is an organic contaminated (high C) sample surface (to be ignored), followed by a 'true' analysis].

The SIMS profile shows a film surface depleted of carbon and hydrogen by the hydrogen plasma treatment. This is a not unsurprising result and is consistent with a measurable difference in the dielectric constant of this

surface layer and the bulk of the film. When this surface is etched away the remainder of the film (adjusted for its reduced thickness) has a lower dielectric constant than the whole of the film including this surface layer. Yet the whole film including this carbon depleted surface has a lower k value than an FTM treated film.

Wet etch rate experiments show that the hydrogen plasma treatment commences at the upper surface and progresses through the film. The hydrogen plasma treated film wet etches considerably more slowly than an FTM treated film and thus provides clear evidence that depth of treatment increases with plasma treatment times.

It is postulated that the hydrogen plasma treatment effectively replaces Si-CH_3 in the film with $\text{Si-CH}_2\text{-Si}$ (by intermediate reactions in which hydrogen ions and radicals play a part) with increased Si-Si linkage responsible for the increase in the refractive index.

CLAIMS

1. A method of forming a film on a substrate comprising:
 - (a) positioning the substrate on a support in a chamber;
 - (b) supplying to the chamber in gaseous or vapour form a
5 silicon-containing organic compound and an oxidising agent in the presence of a plasma to deposit a film on the substrate; and
 - (c) setting the film such that carbon-containing groups are contained therein.
- 10 2. A method according to claim 1, wherein the oxidising agent is oxygen.
3. A method according to claim 1 or 2, wherein the silicon-containing organic compound is an alkylsilane.
4. A method according to any preceding claim, herein the
15 silicon-containing organic compound is a tetraalkylsilane.
5. A method according to claim 4, wherein the silicon-containing organic compound is tetramethylsilane.
6. A method according to claim 1 or 2, wherein the silicon-containing organic compound is a methylsilane.
- 20 7. A method according to claim 3, wherein the silicon-containing organic compound is cyclohexyldimethoxymethylsilane.

8. A method according to any preceding claim, wherein the film is deposited on a substrate positioned on a low temperature support.

9. A method according to claim 6, wherein the support is at a temperature between about 0°C to about 60°C.

10. A method according to claim 6 or claim 7 wherein the support is at about 30°C,

11. A method according to any preceding claim, further comprising providing a plasma during deposition of the film.

12. A method according to any preceding claim, wherein the set film has a dielectric constant of about 2.55 or less.

13. A method of forming a film on a substrate comprising:

(a) positioning the substrate on a support in a chamber.

(b) supplying to the chamber in gaseous or vapour form tetramethylsilane and oxygen in the presence of a plasma to deposit a film on the substrate; and

(c) setting the film such that carbon-containing groups are contained therein.

14. A method according to any preceding claim, further comprising supplying the plasma from an RF power source connected to an electrode opposing the substrate support.

15. A method according to any preceding claim wherein the substrate support is at D.C. ground during the application of the plasma.
- 5 16. A method as claimed in any one of the preceding claims wherein the film is set by exposing it to an H₂ containing plasma without any prior annealing or heating step.
17. A method as claimed in claim 16 wherein the H₂ containing plasma is substantially only a H₂ plasma.
- 10 18. A method as claimed in claim 16 or 17 wherein the H₂ containing plasma treatment last for between 30 seconds and 30 minutes.
- 15 19. A method as claimed in claim 16 or 17 wherein the H₂ containing plasma treatment lasts from 1 to 10 minutes.
20. A method as claimed in claim 16 or 17 wherein the H₂ containing plasma treatment step lasts no more than 5 minutes.
- 20 21. A method as claimed in claim 16 or 17 wherein the H₂ containing plasma treatment step lasts no more than 10 minutes.
22. A method as claimed in claim 16 where the hydrogen containing plasma is applied simultaneously with heating.

23. A method as claimed in claim 22 where the substrate is heated to approximately 400°C.

24. A method substantially as hereinbefore described with reference to the accompanying drawings and examples.

5 25. A method as claimed in claim 1 where the setting of the film substantially removes water and/or OH peaks from the FTIR spectra of the as deposited film.

10 26. An apparatus for forming a film on a substrate, the apparatus comprising:

- (a) a support for the substrate positioned in a chamber;
- (b) means for supplying to the chamber in gaseous or vapour form a silicon-containing organic compound and an oxidising agent in the presence of a plasma to
15 deposit a film on the substrate; and
- (c) means for setting the film such that carbon-containing groups are contained therein.

20 27. An apparatus according to claim 26, further comprising means for improving the uniformity of the deposition of the film on the substrate.

28. An apparatus according to claim 27, wherein the means for improving the uniformity is arranged around a showerhead.

29. An apparatus substantially as hereinbefore described with reference to, and as illustrated in, the accompanying drawings.

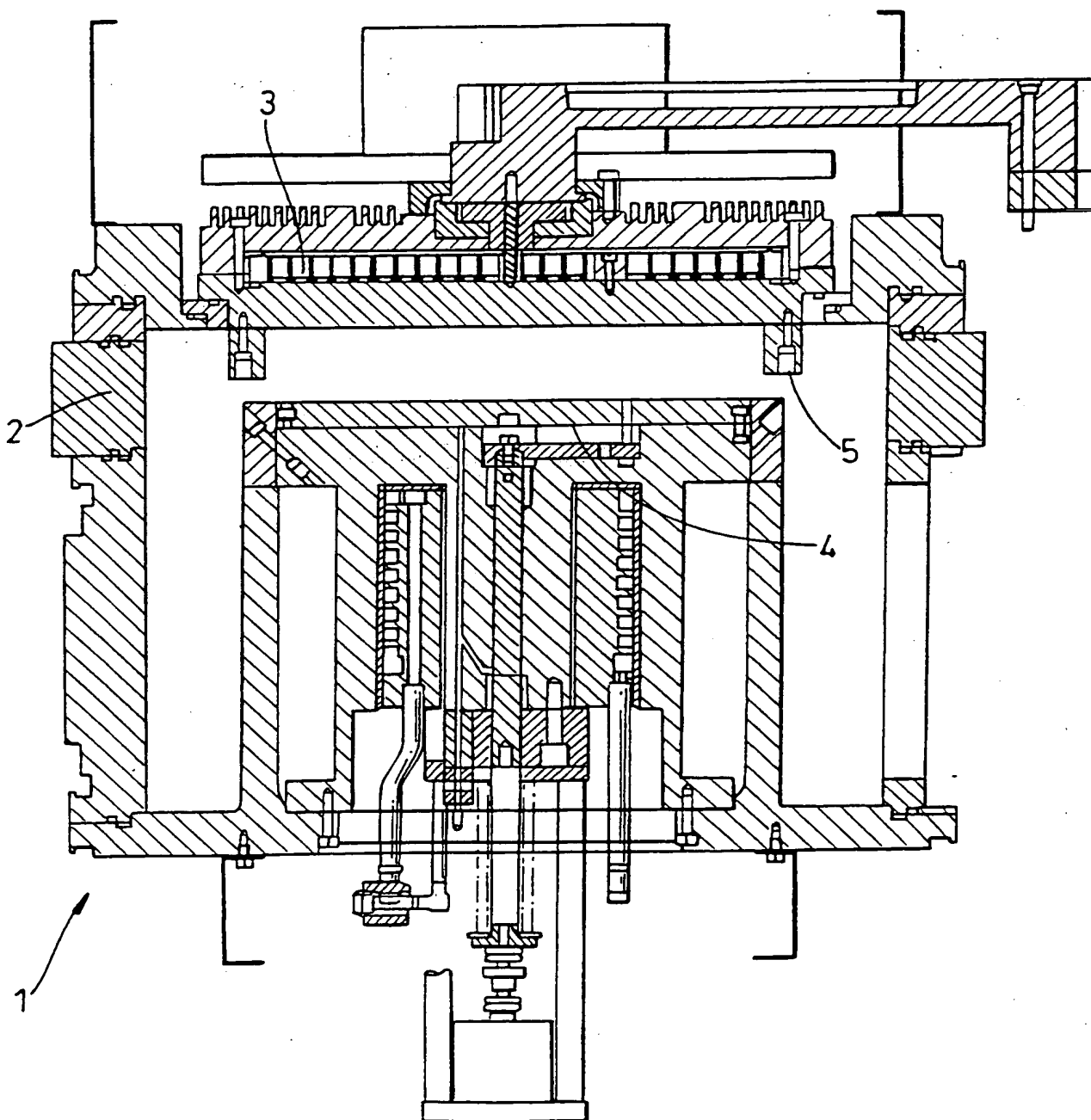
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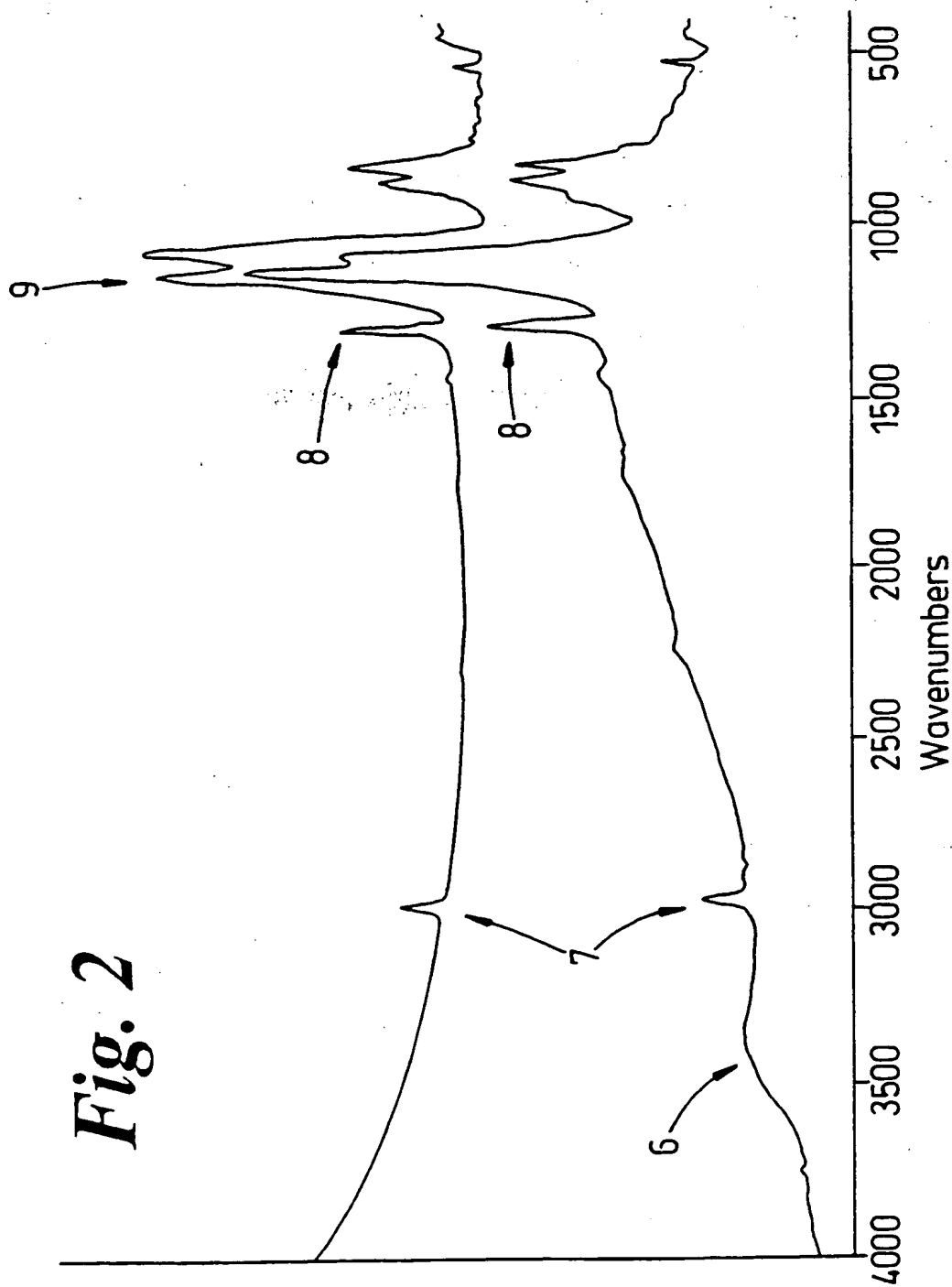
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*Fig 1*

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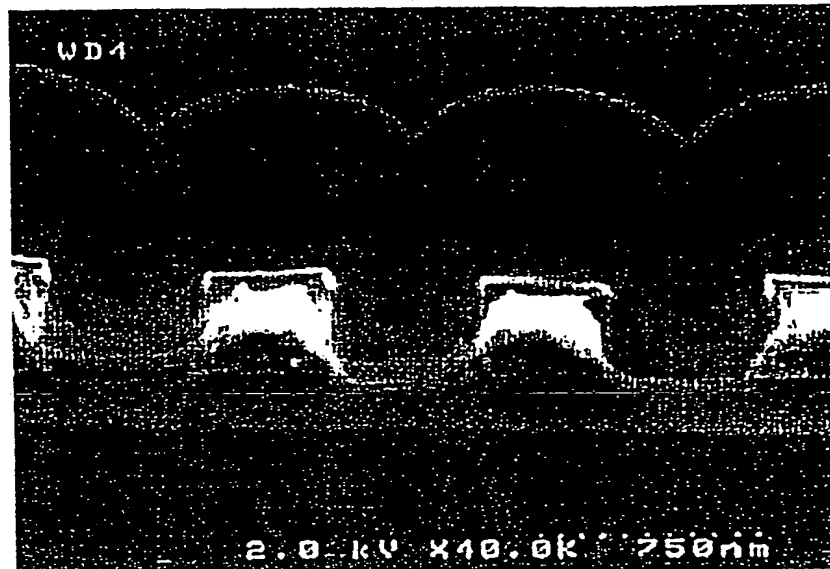
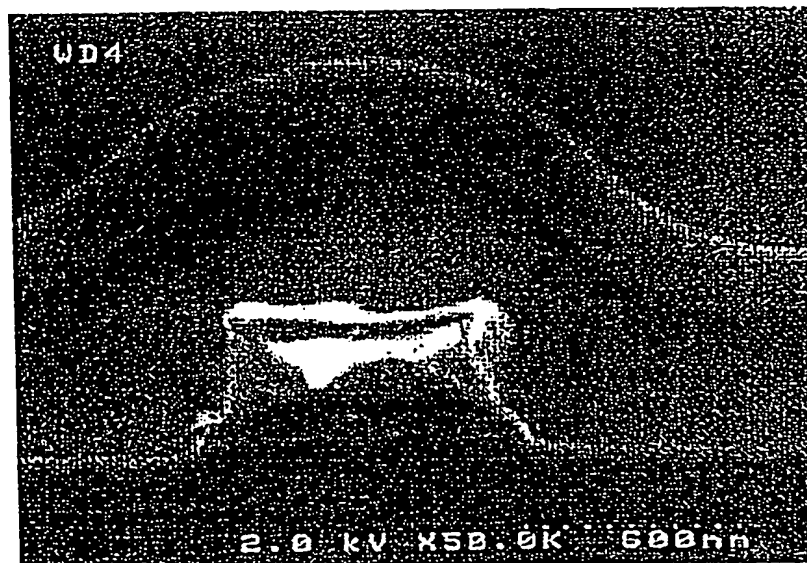
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210 / 200 TMS/O₂ 2000MT 250 W 0°C PLATEN PRE AND POST ANNEAL

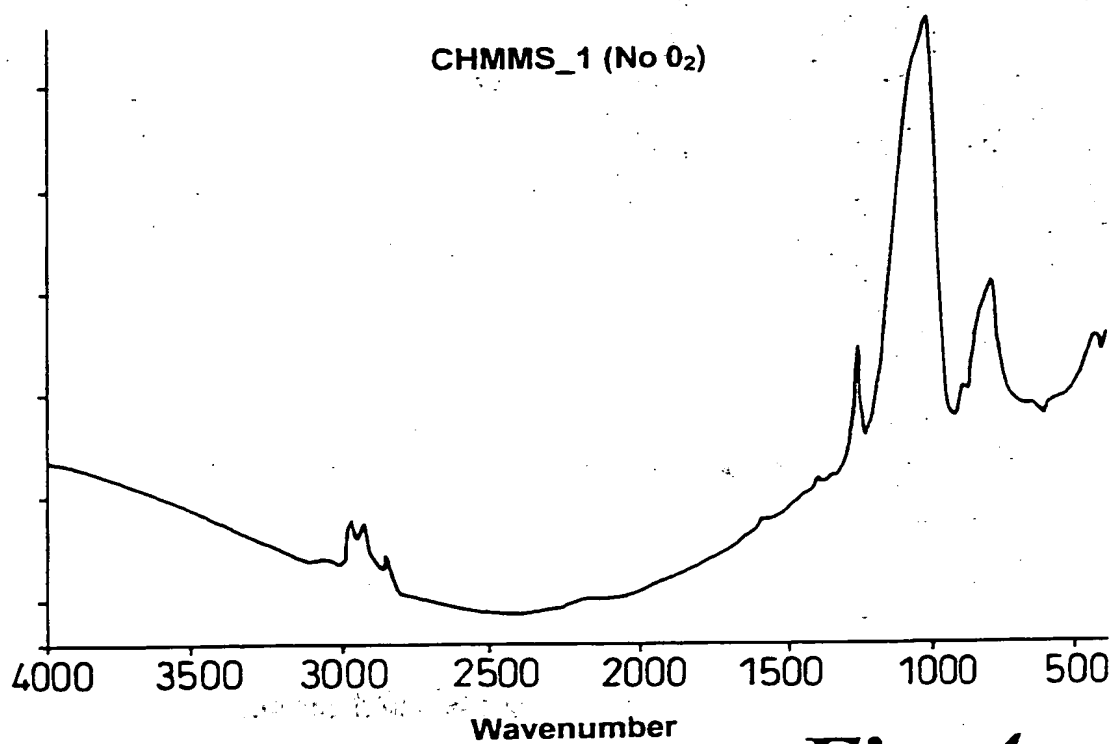
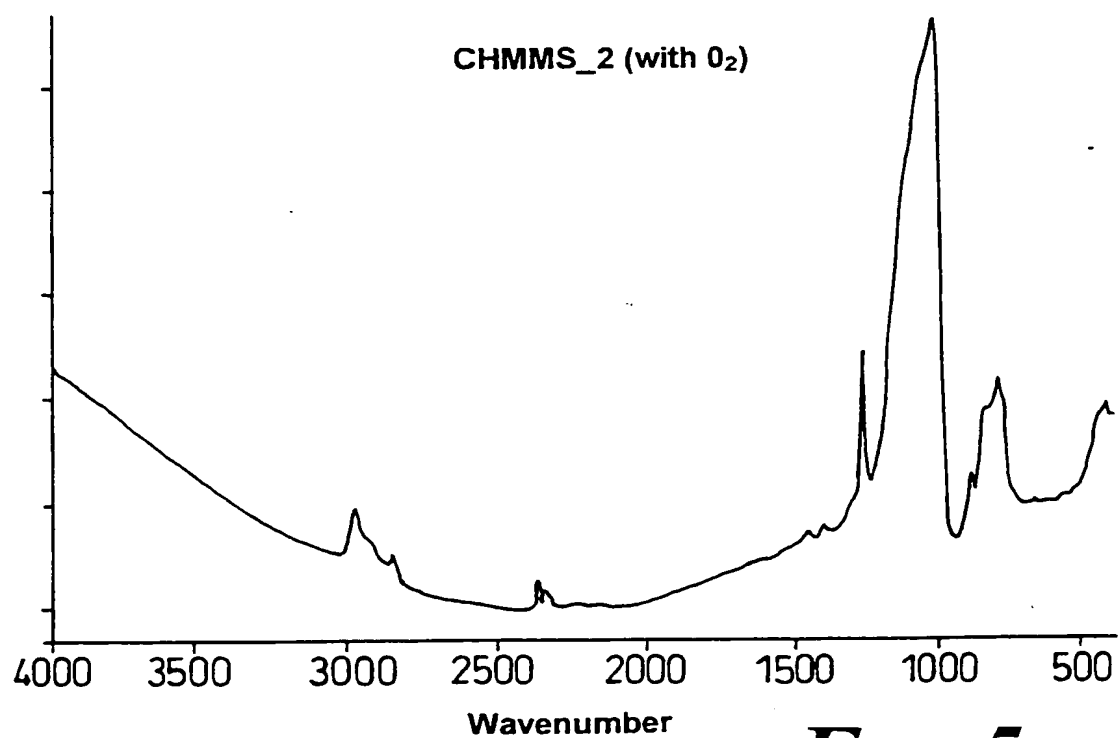
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*Fig. 3(a)**Fig. 3(b)*

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*Fig. 4**Fig 5*

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Date	Run No	CHMMS	H ₂ O ₂	O ₂	N ₂	RF Power (W)	Pressure (mT)	Platen (°C)	S/head (°C)	Dep rate (Å/min)	Uniformity	Rj	Rj Range	SiC/SiO	SiH/SiO	CH/SiO	K
10/05/99	1	1000	0.75	0	0	50	1500	70	100	1166	6.6	1.482	0.0007				
	2	1300	0.75	0	0	50	900	70	100	681	5.1	1.3702	0.0074				
	3	1300	0.7	0	0	100	900	70	100	2542	6.5	1.3321	0.01				
	4	1300	0.7	0	0	100	900	70	100	1853	6.1	1.3676	0.015				
	6	1300	0.7	0	0	100	900	70	100	1450	8.5	1.3498	0.0125				
	7	1300	0.7	0	0	100	900	70	100	3916	8.1	1.4736	0.0023				
	8	1300	0.7	0	0	100	900	70	100	2008	8.3	1.3587	0.0075				
	9	1300	0.7	0	0	100	900	0	100	3965	22.1	1.5007	0.005				
	10	1300	0.7	0	0	200	900	5	100	2097	22.5	1.3749	0.005				
	11	1300	0.7	0	0	200	900	0	100	1392	13.5	1.4871	0.005				
										716	13.9	1.374	0.025	0.0343	0	0.1062	
Pot Refilled 24/05/99	12	800	0.5	0	0	100	900	70	100	1176	27.3	1.4831	0.0163	0.0183	0.0063	0.1102	
	13	800	0.4	0	0	500	900	70	100	738	25.6	1.4064	0.0156	0.0191	0	0.0069	
	14	800	0.4	0	0	250	900	70	100	2000							
	15	800	0	0	0	500	900	70	100	1731	15.9	1.4618					
	16	800	0	0	0	250	900	70	100	9938	35	1.458					
	17	800	0	0	0	500	900	70	100	2166	16.2	1.4569					
	18	800	0	0	0	250	900	70	100	-10000	-30	1.45					
	19	800	0	0	0	250	900	70	100	-6000	-45						2.55
	20	800	0	0	0	250	900	40	100	5200							2.5-2.7
	21	800	0	0	0	250	900	40	100	7200	23.4	1.4938					
	22	800	0	0	0	250	900	20	100	5338				0.03	0.039	0.2353	3.2
	23	800	0	0	0	250	900	20	100	4200	14.4	1.4913	0.015	0.0396	0.027	0.237	2.87

Fig. 6

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Flowfill chamber depositions using Cyclohexyldimethoxymethylsilane

P727 - Flowfill chamber (Flow_1), 40mm electrode gap - Syringe delivery system

Process Parameter**Bulk Film Properties**

Run Number	CHMMS (g/min)	O ₂ Flow (sccm)	N ₂ Flow (sccm)	Pressure (mT)	Power (W)	Showerhead (°C)	Platen (°C)	Dep rate (Å/min)	Uniformity (%)	Refractive Index	Dielectric Constant	FTIR P.A.R.		Comment
												SiC/SiO ₂	SiH/SiO ₂	CH/SiO ₂
1	0.65	0	0	900	250	100	50	7224	10.6	1.4819		0.0357	0.0111	0.1347
2	0.65	0	0	900	250	100	50					0.0345	0.0296	0.03672
3	0.65	0	100(1+8)	900	250	100	50	8190	6.5	1.4875		0.039	0.015	0.1545
4	0.65	0	50(1+8)	900	250	100	50	5810	22.8	1.4852		0.0354	0.0269	0.3499
5	0.65	0	50(1+8)	900	250	100	50	8289	13.1	1.51		0.0384	0.0107	0.131
6	0.65	0	50(1+8)	900	250	100	50	8908	4.47	1.5089		0.0378	0.0139	0.1238
7	0.65	0	100(1+8)	900	250	100	50	8717	2.37	1.4475		0.0384	0.0172	0.1886
8	0.65	0	100(1+8)	900	100	100	50	2789	9.2	1.4775		0.0349	0.0367	0.3518
9	0.65	0	100(1+8)	900	500	100	50	12748	3.4	1.489				
10	0.65	0	100(1+8)	900	500	100	50	14222	1.77	1.538				
11	0.85	0	200(1)	900	500	100	50	14192	1.5	1.5228				
12	0.85	0	200(8)	900	500	100	50	14282	1	1.5444				
13	0.85	0	100(1+8)	500	500	100	50	9780	3.7	1.4895				
14	0.85	0	100(1+8)	500	500	100	50	11382	6.2	1.4688				
15	0.85	0	200(8)	900	500	100	50	18116	5.6	1.4634				
16	0.85	0	100(1+8)	900	250	100	50	10242	6.6	1.4558				
17	0.65	0	100(1+8)	900	250	100	50				3*			
18	0.85	0	200(8)	900	500	100	50				2.78*			
19	0.85	0	100(1+8)	500	500	100	50				2.82*			
20	0.65	0	100(1+8)	900	500	100	50				3.01*			
21	0.85	0	100(1+8)	900	500	100	50	7869	7.8	1.5144				
22	0.85	0	100(1+8)	900	500	100	50	15697	5.7	1.5387				
23	0.85	0	100(1+8)	900	500	100	50	14751	3.5	1.4737				
24	0.85	0	100(1+8)	900	500	100	50	14345	0.9	1.4737				
25	0.85	0	100(1+8)	900	500	100	50	14079	1.6	1.4882				
26	0.85	0	200(8)	900	500	100	50	18864	4.5	1.4332				
27	0.85	0	200(8)	900	750	100	50	17841	7.2	1.4327				
28	0.85	0	200(8)	900	250	100	50	11511	6.3	1.4263				
29	0.85	0	200(8)	900	500	100	50	15565	3.5	1.4856				
30	0.85	0	200(8)	900	500	100	50	14807	3.1	1.4575		0.0317	0.0193	0.1366
31	0.85	0	200(8)	900	750	100	50	16998	3.8	1.503		0.0336	0.0096	0.0785
32	0.85	0	200(8)	900	250	100	50	11658	11.5	1.499		0.0284	0.018	0.1418
33	0.85	0	200(8)	900	500	100	50				2.56	0.0342	0.0338	0.3437
34	0.85	0	200(8)	900	500	100	50				2.66			
35	0.85	0	200(8)	900	500	100	50	17106	3.7	1.4552		0.0309	0.0199	0.1562
36	0.85	0	200(8)	900	500	100	50	17194	3.7	1.458		0.031	0.0202	0.1498
37	0.85	0	200(8)	1200	900	100	50	24708	2.2	1.5316		0.0311	0.0198	0.1338
38	0.85	0	200(8)	600	500	100	50	9953	1	1.5109		0.0353	0.018	0.1283
39	0.85	0	200(8)	600	250	100	50	7128	0.87	1.5296		0.035	0.0248	0.1258
40	0.85	50	150(8)	900	250	100	50	9652	3.8	1.4575		0.0285	0.0162	0.2006
41	0.85	50	150(8)	900	500	100	50	18448	3.8	1.4209		0.0203	0.009	0.0839

Fig 7 (Part 1 of 3)

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Run Number	CHMMS (g/min)	O ₂ Flow (sccm)	N ₂ Flow (sccm)	Pressure (mT)	Power (W)	Showerhead (°C)	Platen (°C)	Dpp rate (Å/min)	Uniformity (%)	Refractive Index	Dielectric Constant	SICISIO	FTIR P.A.R. SHISIO	CHISIO	Comment
42	0.85	50	250(8)	900	500	100	50	~1.8µm	-	-	-	0.0209	0.0058	0.0572	Entire film cloudy
43	0.85	200	0	900	500	100	50	17888	5.2	1.3978	-	0.0161	0.003	0.0356	Powder stripes
44	0.85	50	150(8)	900	1000	100	50	17896	6.4	1.5031	-	0.0145	0.0068	0.0588	Hazy powder
45	0.85	0	200(8)	600	250	100	50	8213	3.4	1.4669	-	0.0378	0.0164	0.2189	RI Water
46	0.85	0	200(8)	600	250	100	50	9912	8.2	1.4849	-	-	-	-	RI Water
47	0.85	0	200(8)	1200	100	100	50	1792	29.2	1.4635	-	-	-	-	RI Water
48	0.85	0	200(8)	600	250	100	50	10233	3.3	ix	-	0.0387	0.0516	0.3823	No FTS
49	0.85	0	200(8)	600	250	100	50	-	-	-	2.56	-	-	-	5min FTS, no cap
50	0.85	0	200(8)	800	250	100	50	-	-	-	2.72	-	-	-	Water thin, 1st of day, 5min FTS, Cap
51	0.85	0	200(8)	900	250	100	50	-	-	-	3.16	-	-	-	No FTS
52	0.85	0	200(8)	900	250	100	50	-	-	-	2.33	-	-	-	30min FTS, Cap
53	0.85	0	200(8)	900	250	100	50	13034	5.7	ix	-	0.0371	0.0532	0.3933	No FTS
54	0.85	0	200(8)	900	250	100	50	13929	5.7	ix	-	0.0367	0.0599	0.3885	No FTS
55	0.85	0	200(8)	900	250	100	50	11131	7.7	ix	-	0.0368	0.0135	0.151	5min FTS
56	0.85	0	200(8)	900	250	100	50	9809	4.9	ix	-	0.0376	0.0101	0.0785	30min FTS
57	0.85	0	200(8)	900	250	100	50	9853	3.4	ix	-	0.0363	0.0082	0.0583	50min FTS
58	0.85	0	200(8)	900	250	100	50	-	-	-	2.62	-	-	-	5min FTS, no cap
59	0.85	0	200(8)	900	250	100	50	-	-	-	2.55	-	-	-	30min FTS, no cap
60	0.85	0	200(8)	900	250	100	50	-	-	-	2.52	-	-	-	60min FTS, no cap
61	0.85	0	200(8)	900	250	100	50	13020	8.1	1.5351	-	0.0374	0.0551	0.3939	No FTS
62	0.85	0	200(8)	900	250	100	50	12801	7.2	1.5658	-	0.0367	0.0573	0.3911	No FTS
63	0.85	0	200(8)	900	250	100	50	12550	6.5	1.4763	-	0.0322	0.02	0.2575	5min FTS @500°C
64	0.85	0	200(8)	900	250	100	50	11885	4.2	1.4466	-	0.0345	0.0126	0.1847	30min FTS @500°C
65	0.85	0	200(8)	900	250	100	50	10879	13.7	1.5553	-	0.0373	0.0102	0.109	60min FTS @500°C
66	0.85	0	200(8)	900	250	100	50	-	-	-	2.83	-	-	-	5min FTS @500°C no cap
67	0.85	0	200(8)	900	250	100	50	-	-	-	2.97	-	-	-	5min FTS, cap @500°C
68	0.85	0	200(8)	900	250	100	50	-	-	-	2.61	-	-	-	60min FTS, cap @500°C
69	0.85	0	200(8)	900	500	100	50	17388	4.2	1.4207	-	0.033	0.0111	0.094	30min FTS
70	0.85	0	200(8)	900	500	100	50	17484	3.8	1.4146	-	0.0323	0.0103	0.0825	60min FTS
71	0.85	0	200(8)	900	500	100	50	-	-	-	2.55	-	-	-	30min FTS, cap
72	0.85	0	200(8)	900	500	100	50	-	-	-	2.53	-	-	-	60min FTS, cap
73	0.85	50	200(8)	900	250	100	50	9869	4.3	1.4218	-	0.0306	0.0073	0.0635	5min FTS (centre cracks)
74	0.85	50	150(8)	900	250	100	50	9564	4.2	1.3767	-	0.0299	0.0064	0.0403	30min FTS no cracks
75	0.85	50	150(8)	900	250	100	50	-	-	-	2.26	-	-	-	30min FTS, cap
76	0.85	0	200(8)	900	250	100	50	13971	8	1.4022	-	-	-	-	RI Water
77	0.85	0	200(8)	900	250	100	50	12355	8.3	1.4308	-	-	-	-	RI Water
78	0.85	50	150(8)	900	250	100	50	11928	6.1	1.4115	-	-	-	-	RI Water
79	0.85	50	150(8)	900	250	100	50	11070	6.9	1.3942	-	-	-	-	RI Water
80	0.85	0	200(8)	900	500	100	50	25340	7.4	1.4493	-	-	-	-	RI Water
81	0.85	0	200(8)	900	500	100	50	22157	6.5	1.4307	-	-	-	-	RI Water
82	0.85	0	200(8)	900	500	100	50	17612	3.4	1.4354	-	-	-	-	5min FTS
83	0.85	0	200(8)	900	500	100	50	17736	2.7	1.5128	-	-	-	-	No FTS
84	0.85	0	200(8)	900	500	100	50	14636	2.4	1.4463	-	-	-	-	30min FTS
85	0.85	0	200(8)	900	500	100	20	21774	6.8	1.5393	-	0.0343	0.3071	0.3071	No FTS
86	0.85	0	200(8)	900	500	100	20	-	-	-	-	0.036	0.079	0.079	30min FTS (peeled off)
87	0.85	0	200(8)	900	500	100	20	-	-	-	-	0.0349	0.0749	0.0749	60min FTS (peeled off)
88	0.85	0	200(8)	900	500	100	20	-	-	-	2.43	-	-	-	-

Fig 7 (Part 2 of 3)

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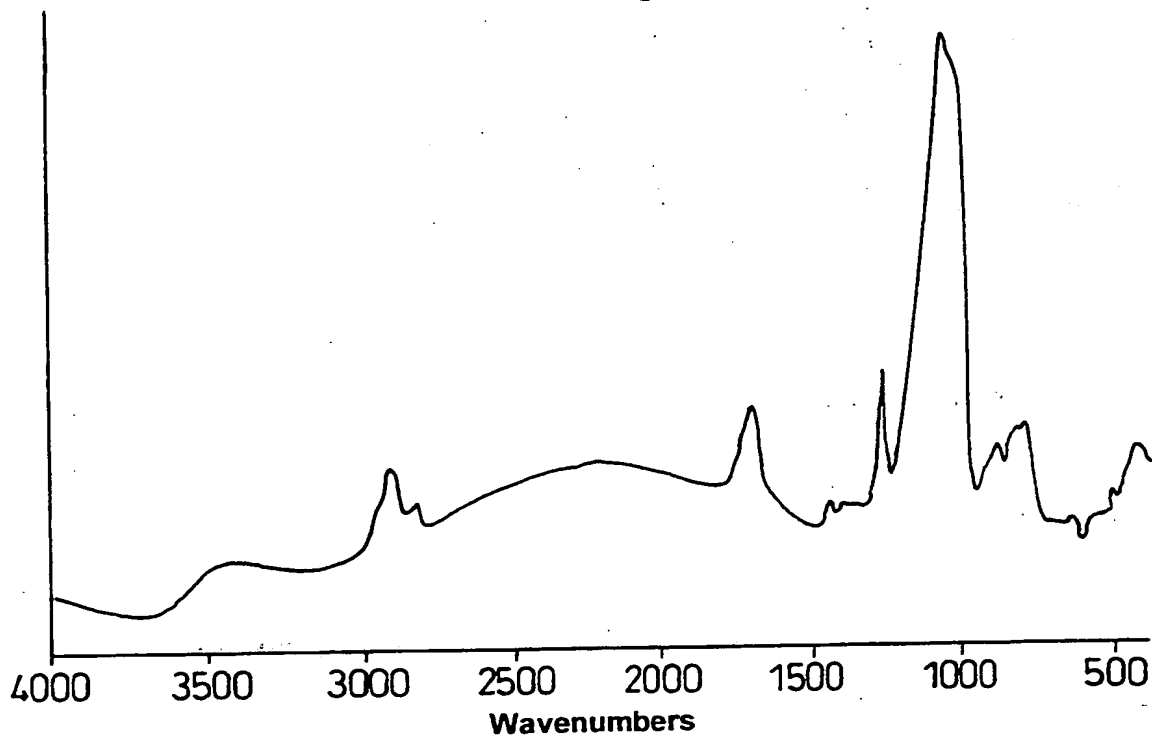
Run Number	CHIMS (g/min)	O ₂ Flow (sccm)	N ₂ Flow (sccm)	Pressure (mT)	Power (W)	Showerhead (°C)	Platen (°C)	Dep rate (Å/min)	Uniformity (%)	Refractive Index	Dielectric Constant	FTIR P.A.R.		Comment
												SIC/SIO	SIHSIO	CH/SIO
89	0.85	0	200(8)	900	500	100	50	17344	2.4	1.4129		0.0341	0.0103	0.1051
90	0.85	25	175(8)	900	500	100	50	17840	1.9	1.4398		0.027	0.0075	0.0776
91	0.85	25	175(8)	900	250	100	50	9278	2.4	1.3875		0.0327	0.0079	0.0725
92	0.85	50	150(8)	900	250	100	50	9863	5.9	1.3646		0.0308	0.0085	0.0456
93	0.85	50	0	900	500	100	50	-10000				0.0344	0.0079	0.0532
94	0.85	50	0	900	500	100	50	21408	4.6	1.4199		0.0273	0.0076	0.0725
95	0.85	50	150(8)	900	250	100	50				2.49			
96	0.85	50	150(8)	900	250	100	50				2.48			
97	0.85	25	175(8)	900	250	100	50	9917	8.8	1.4521		0.0342	0.0093	0.1091
98	0.85	25	175(8)	900	250	100	50	9848	11.7	1.4592		0.0334	0.0117	0.1441
99	0.85	25	175(8)	900	250	100	50				2.437			
100	0.85	25	175(8)	900	250	100	50				2.286			
101	0.85	25	175(8)	900	250	100	50				2.426			
102	0.43	0	100(8)	900	500	100	50	12080	7.6	1.5107		0.0239	0.0093	0.098
103	0.43	0	100(8)	900	500	100	50	12502	5.1	1.5081		0.025	0.0119	0.0116
104	0.64	0	150(8)	900	500	100	50	20470			2.9			
105	0.85	0	400(8)	900	750	100	50	14074	3.5	1.4983		0.0282	0.0149	0.0912
106	0.85	0	400(8)	900	750	100	50	13930	2.4	1.496		0.0278	0.0143	0.0771
107	0.85	0	400(8)	900	750	100	50				2.72			
CHAMBER SPACING CHANGED TO 20MM														
108	0.85	0	200(8)	900	500	100	50	17626		1.3437				
109	0.85	0	200(8)	900	500	100	50	21765	8.4	1.3554				30min FTS
110	0.85	25	175(8)	900	250	100	50	11436	17.6	1.3713				30min FTS, RI Wafer
111	0.85	25	175(8)	900	250	100	50	12828	13.6	1.3888				30min FTS
112	0.85	0	200(8)	900	500	100	50	14280	11.1	1.447				30min FTS, RI Wafer
113	0.85	0	200(8)	900	500	100	50	12185	3.4	1.3756				30min FTS
114	0.85	0	200(8)	400	500	100	50	9049	2.8	1.4745				30min FTS
115	0.85	0	200(8)	400	500	100	50	10620	8.6	1.4549				30min FTS, RI Wafer
116	0.85	0	200(8)	400	500	100	50	9073	3.5	1.4524		0.0255	0.0143	0.0724
117	0.85	0	200(8)	900	500	100	50	14852	2.3	1.4384		0.0343	0.0096	0.0865
118	0.85	0	200(8)	900	500	100	50				2.556			
119	0.85	0	200(8)	400	500	100	50				2.76			
120	0.85	0	200(8)	900	500	100	50	11633		1.4334		0.0363	0.0094	0.0751
121	0.85	0	200(8)	900	250	100	50	8813		1.399		0.0386	0.0091	0.01019
122	0.85	25	175(8)	900	250	100	50	9207		1.3954		0.0329	0.0069	0.0493
123	0.85	50	150(8)	900	250	100	50	10515		1.381		0.0313	0.007	0.0502
124	0.85	15	185(8)	900	250	100	50	10640		1.3807		0.034	0.0051	0.0556
125	0.85	75	125(8)	900	250	100	50	11727		1.3597		0.0295	0.0042	0.0344
126	0.85	100	100(8)	900	250	100	50	12598		1.3481		0.0293	0.0056	0.0336
127	0.85	25	175(8)	900	250	100	50	9208		1.3718		0.0331	0.0061	0.0466
128	0.85	15	185(8)	900	250	100	50				2.414			
129	0.85	75	125(8)	900	250	100	50				2.4			
130	0.85	100	100(8)	900	250	100	50				2.49			
131	0.85	25	175(8)	900	250	100	50				2.41			
132	0.85	35	165(8)	900	250	100	50				2.48			
133	0.85	15	185(8)	900	250	100	50				2.43			
134	0.85	30	170(8)	900	250	100	50				2.45			

Fig. 7 (Part 3 of 3)

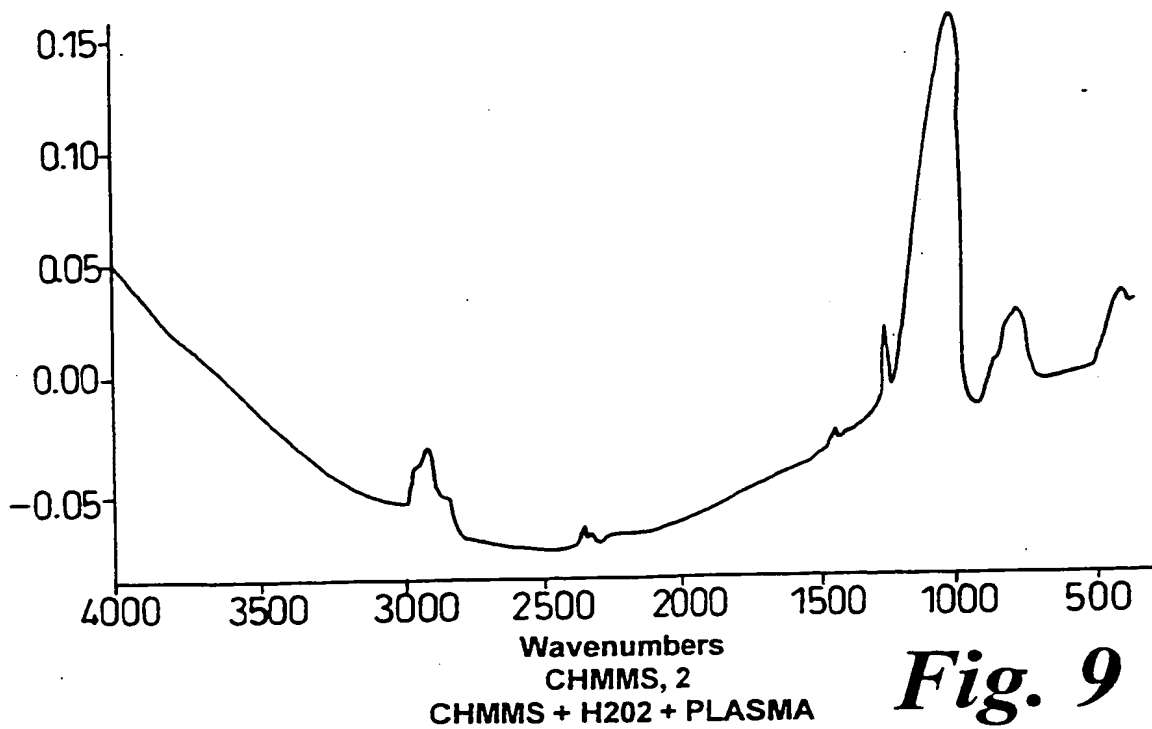
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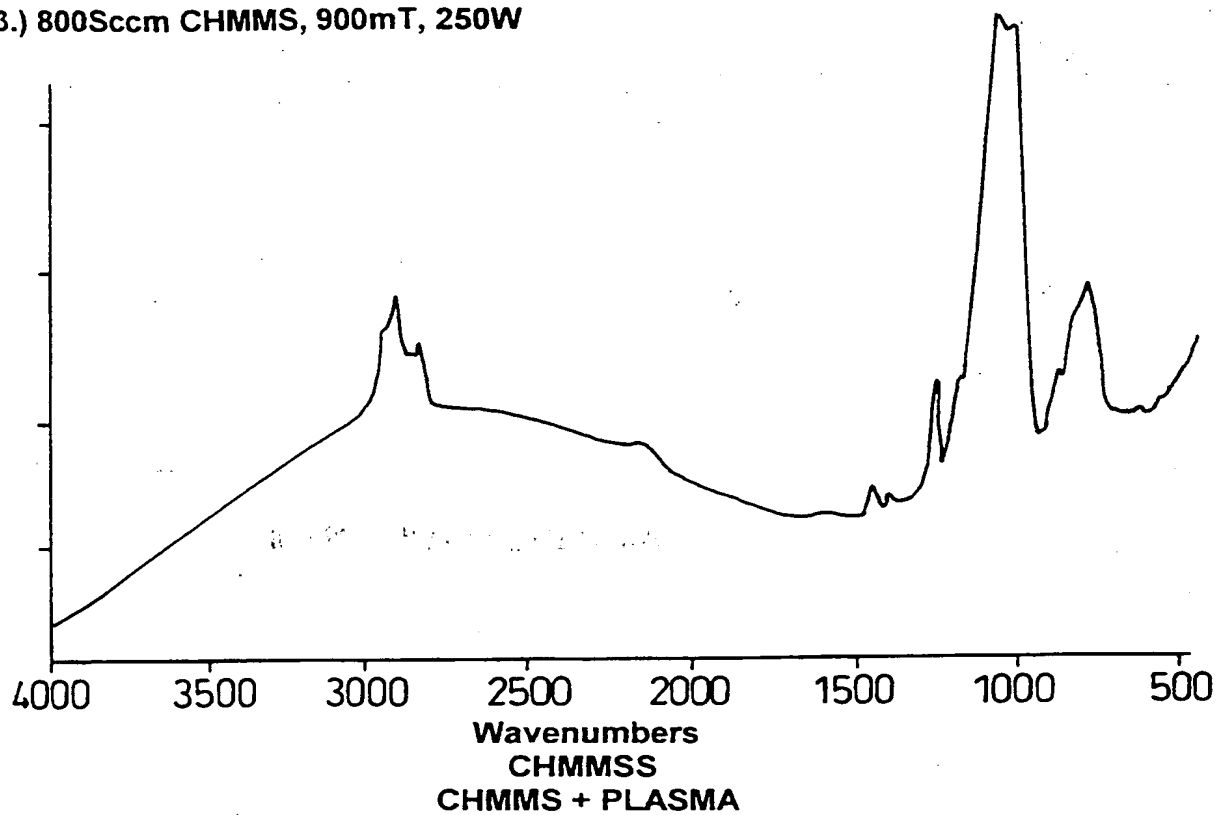
1.) 800CHMMS, 0.4g/min H2O2, 900mT, 250W as deposited

*Fig. 8*

2.) 800Sccm CHMMS, 0.4g/min H2O2, 900mT, 500W

*Fig. 9*

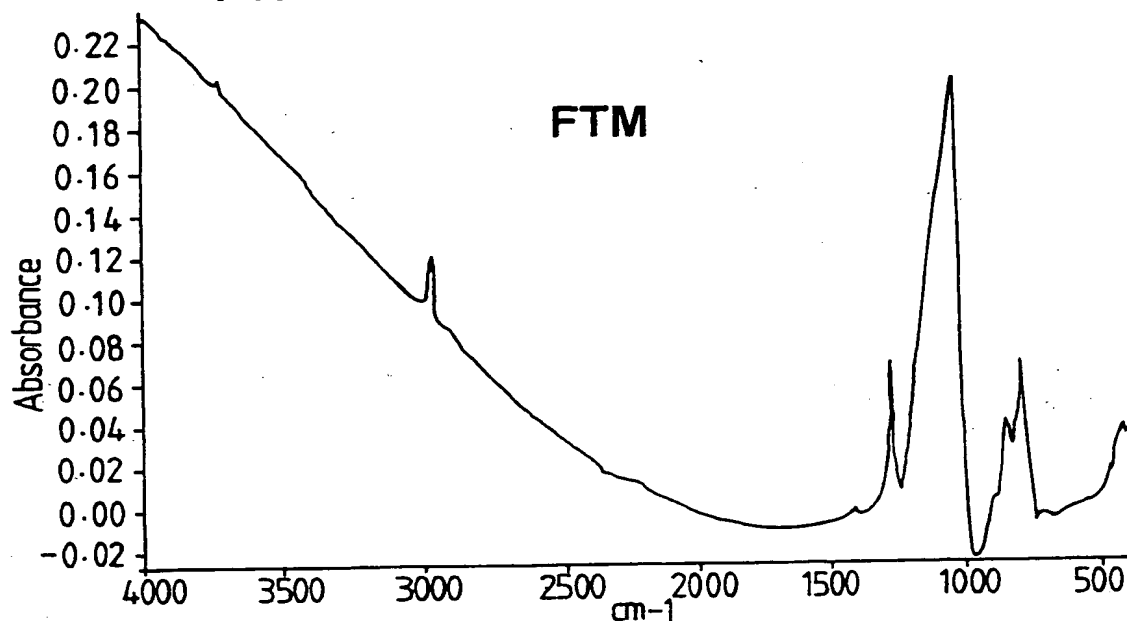
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10/19**3.) 800Sccm CHMMS, 900mT, 250W*****Fig. 10***

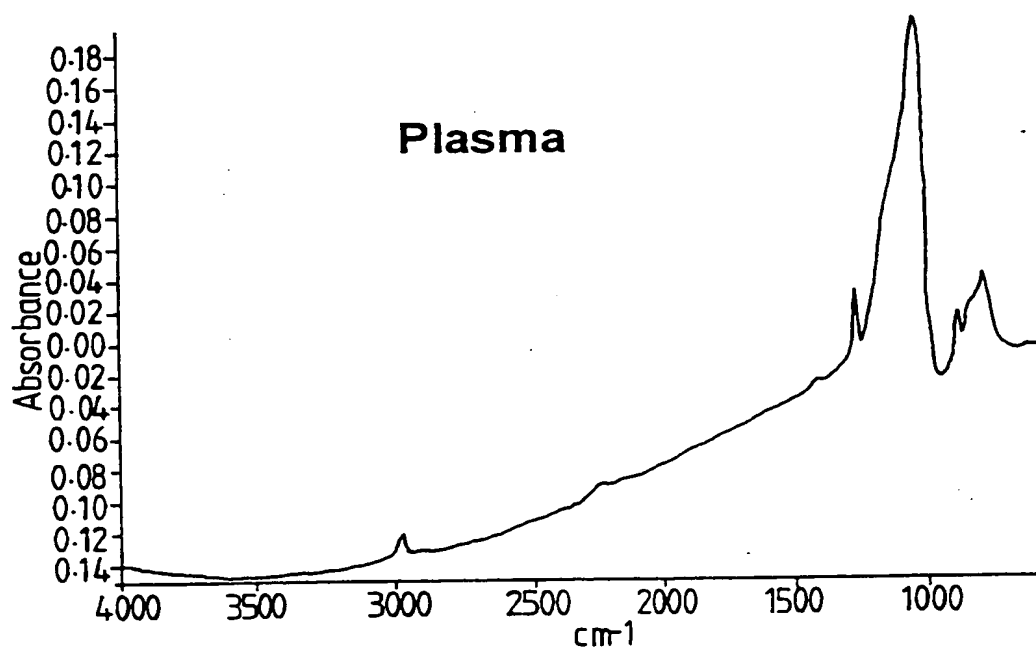
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TMS / O₂ Process
FTM v Plasma Treatment – FTIR



SiC/SiO :	0.0504
SiH/SiO :	0.0279
CH/SiO :	0.0279



SiC/SiO :	0.026
SiH/SiO :	0.019
CH/SiO :	0.0220

Fig 11

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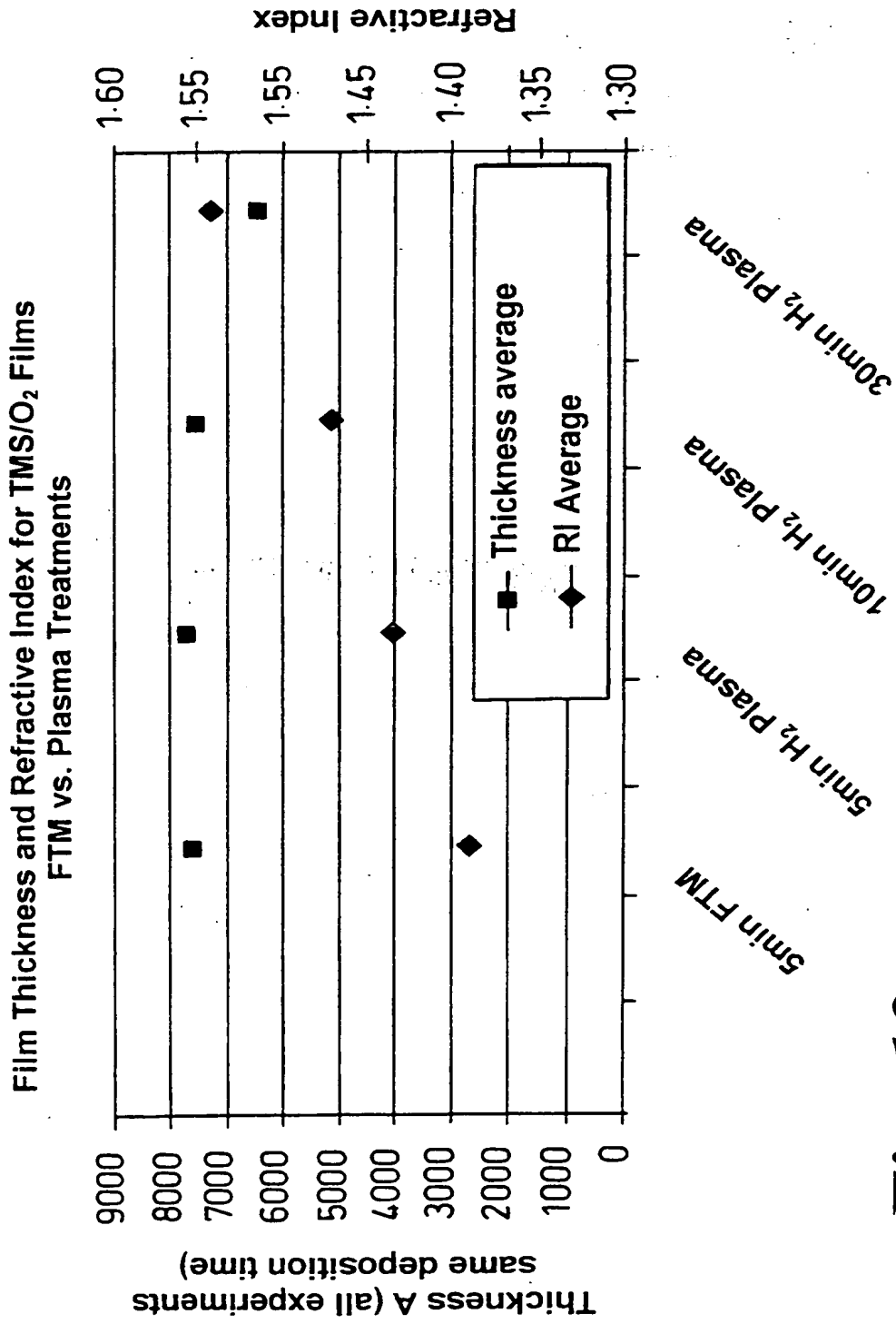
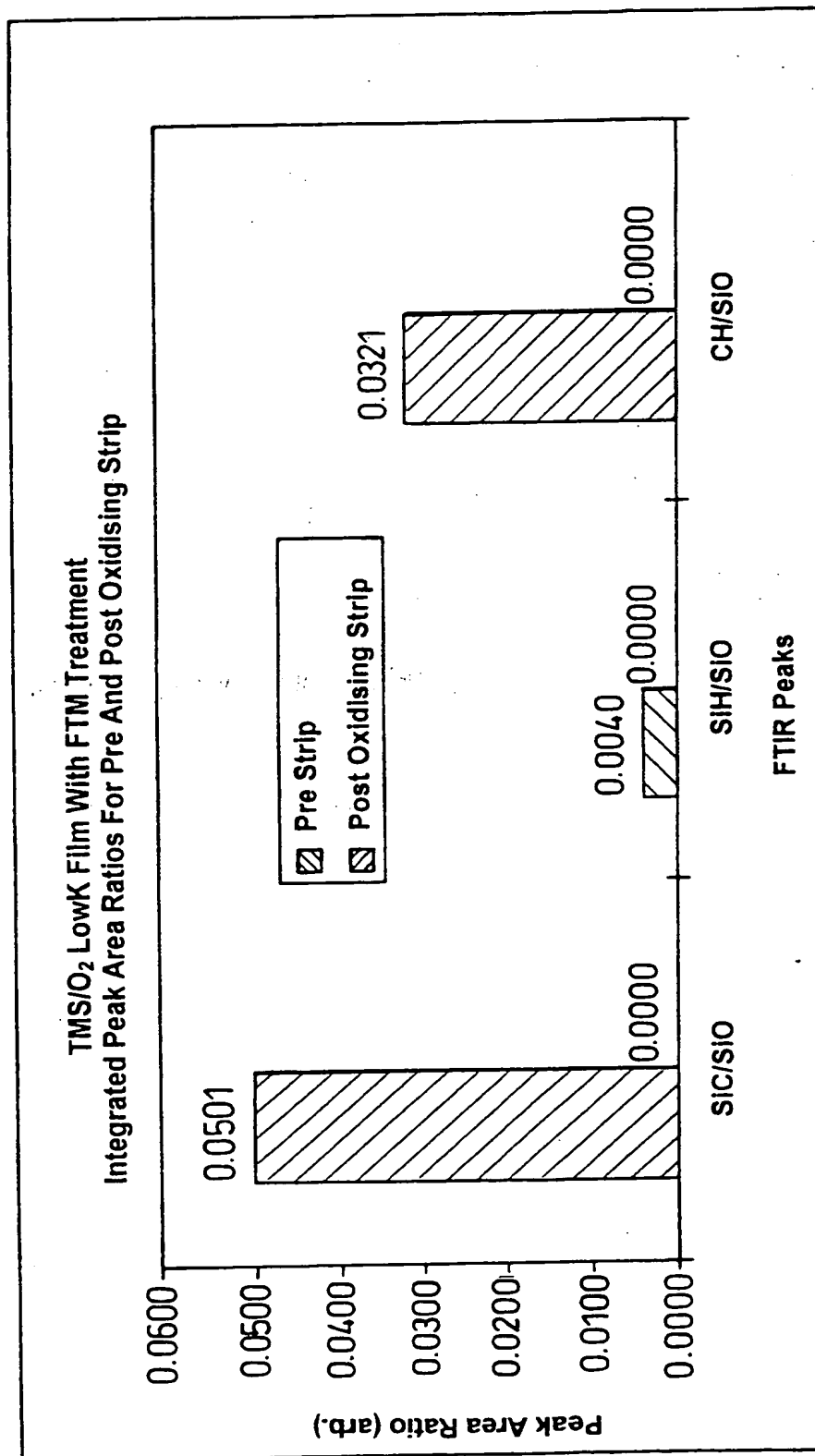


Fig. 12

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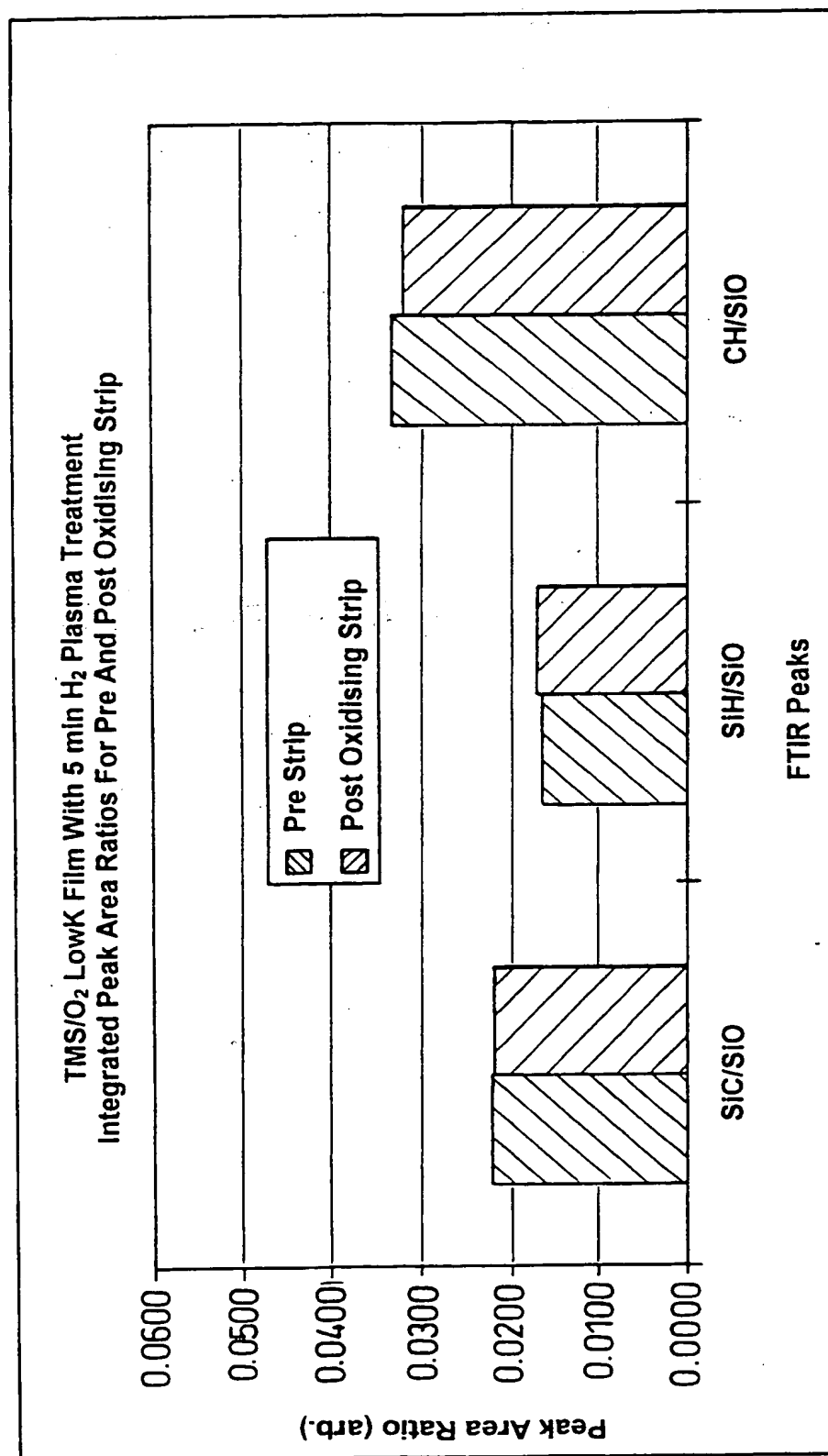
**TMS / O₂ Process
FTM Treatment – FTIR – oxidising strip resistance**

*Fig 13*

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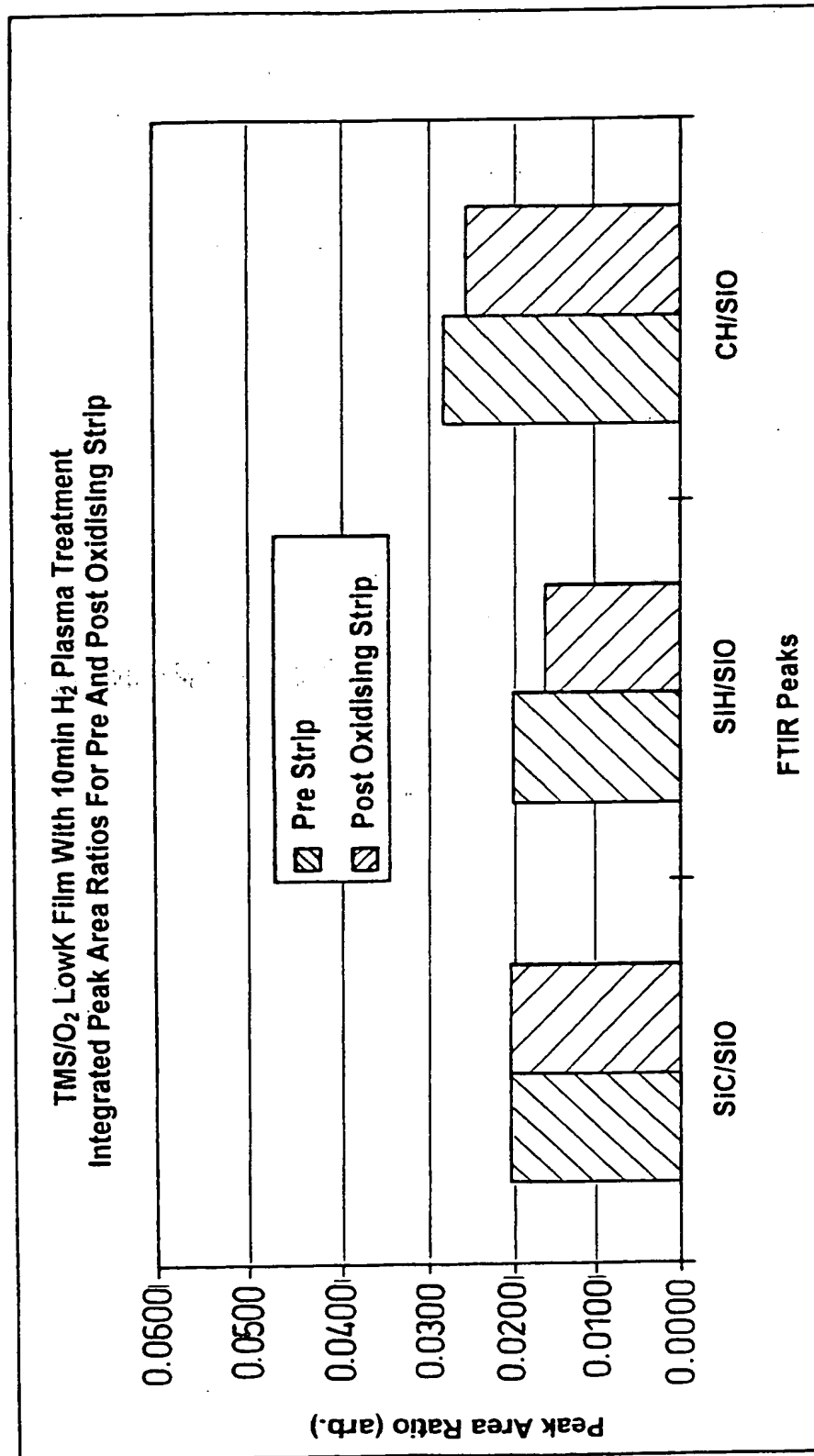
TMS / O₂ Process FTM Treatment – FTIR – oxidising strip resistance

*Fig. 14*

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TMS / O₂ Process FTM Treatment – FTIR – oxidising strip resistance

*Fig. 15*

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TMS / O₂ Process FTM v Treatment stress – oxidising strip resistance

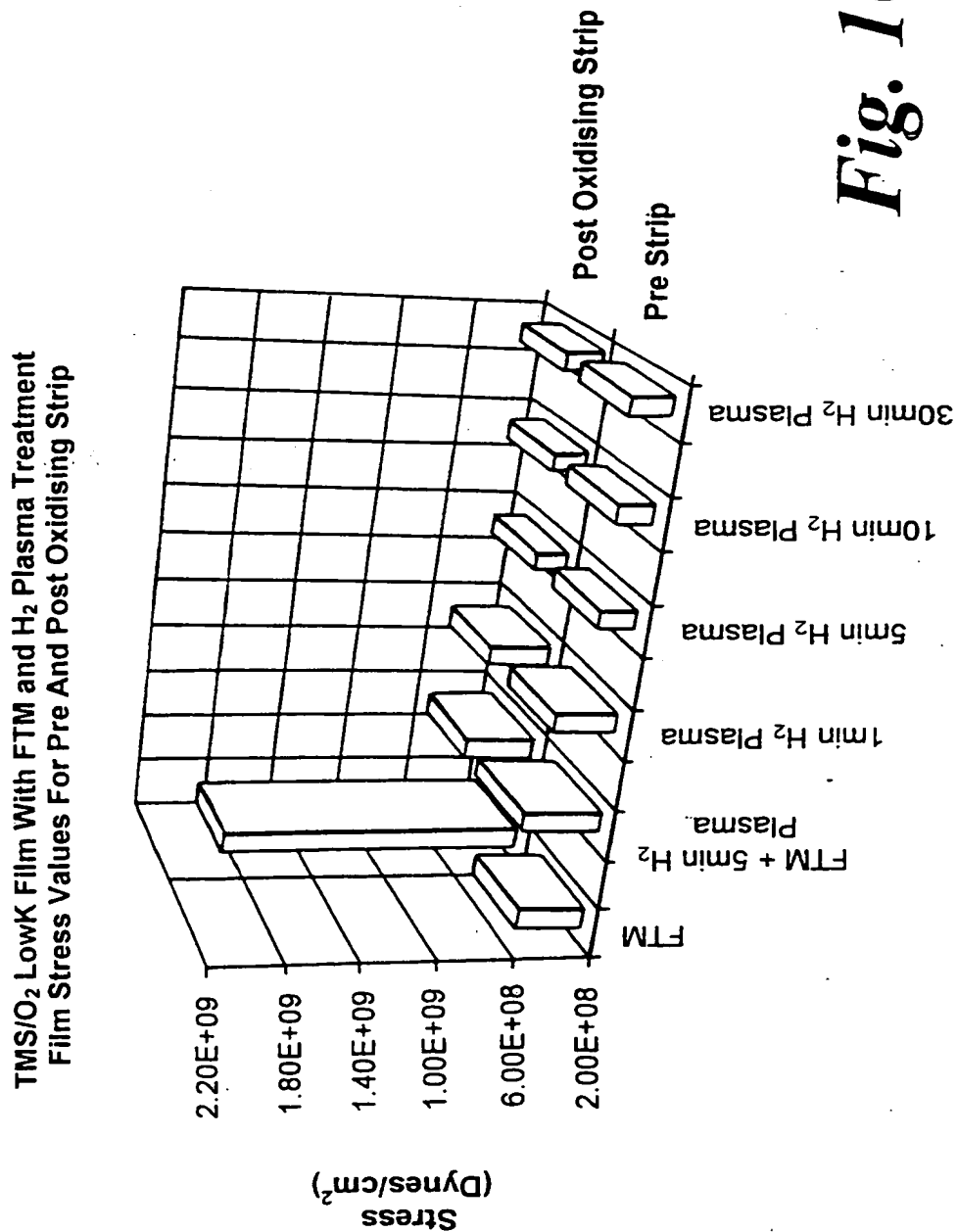


Fig. 16

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TMS / O₂ Process Plasma Treatment – Dielectric constant

Dielectric Constant Values For TMS/O₂ Films
FTM and H₂ Plasma Treatments

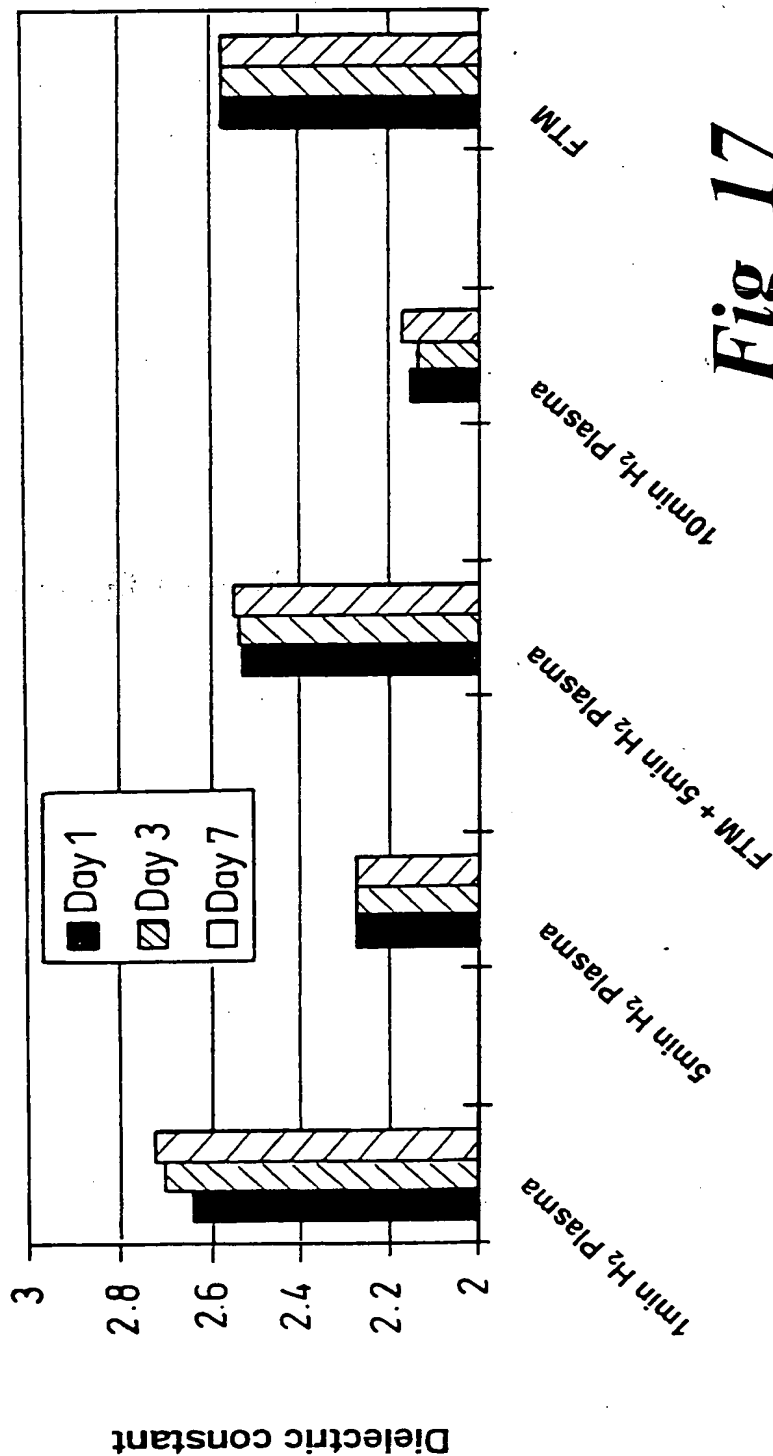


Fig 17

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Dielectric Constant For TMS/O₂ Films
FTM and H₂ Plasma Treatments

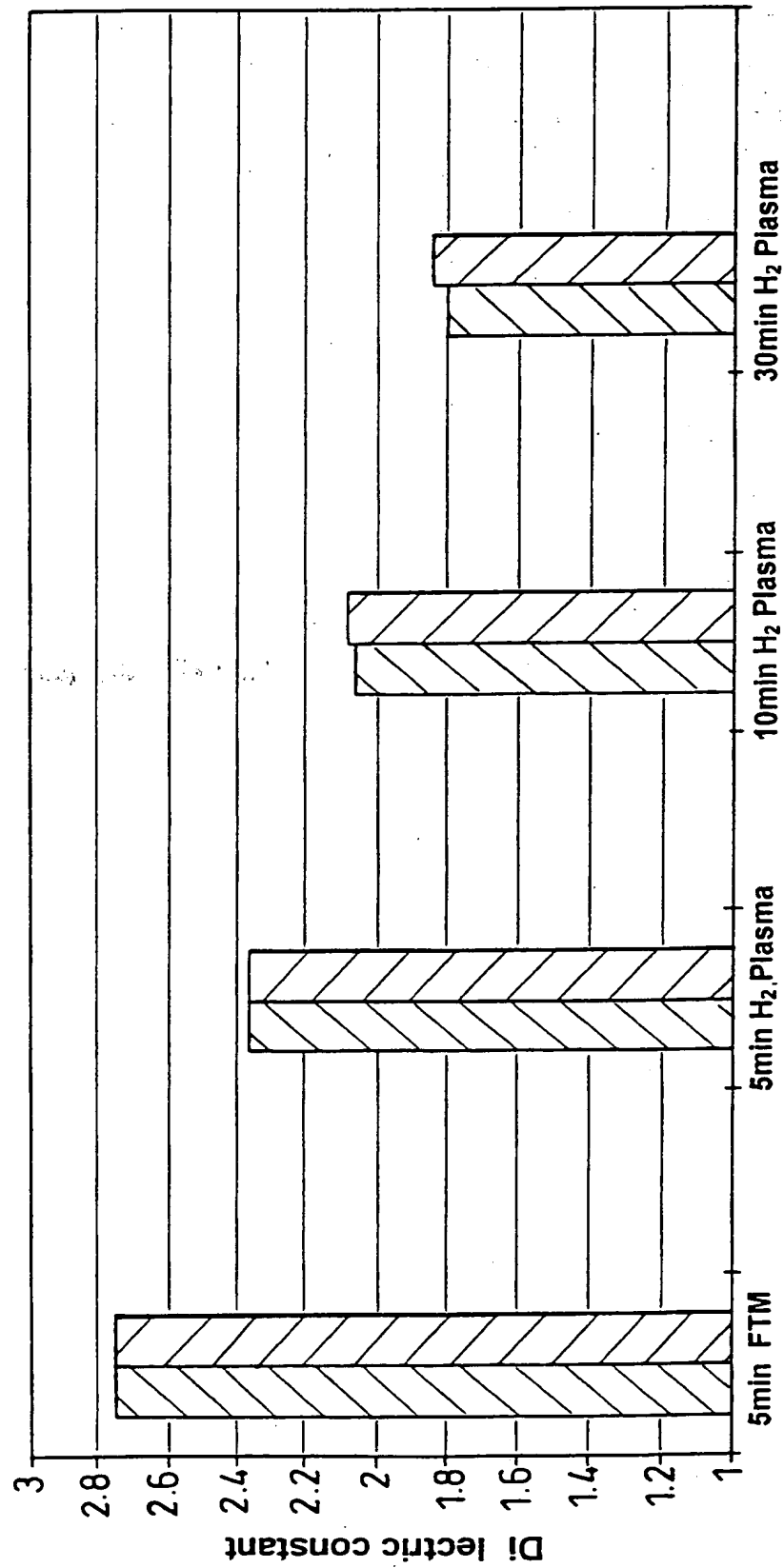
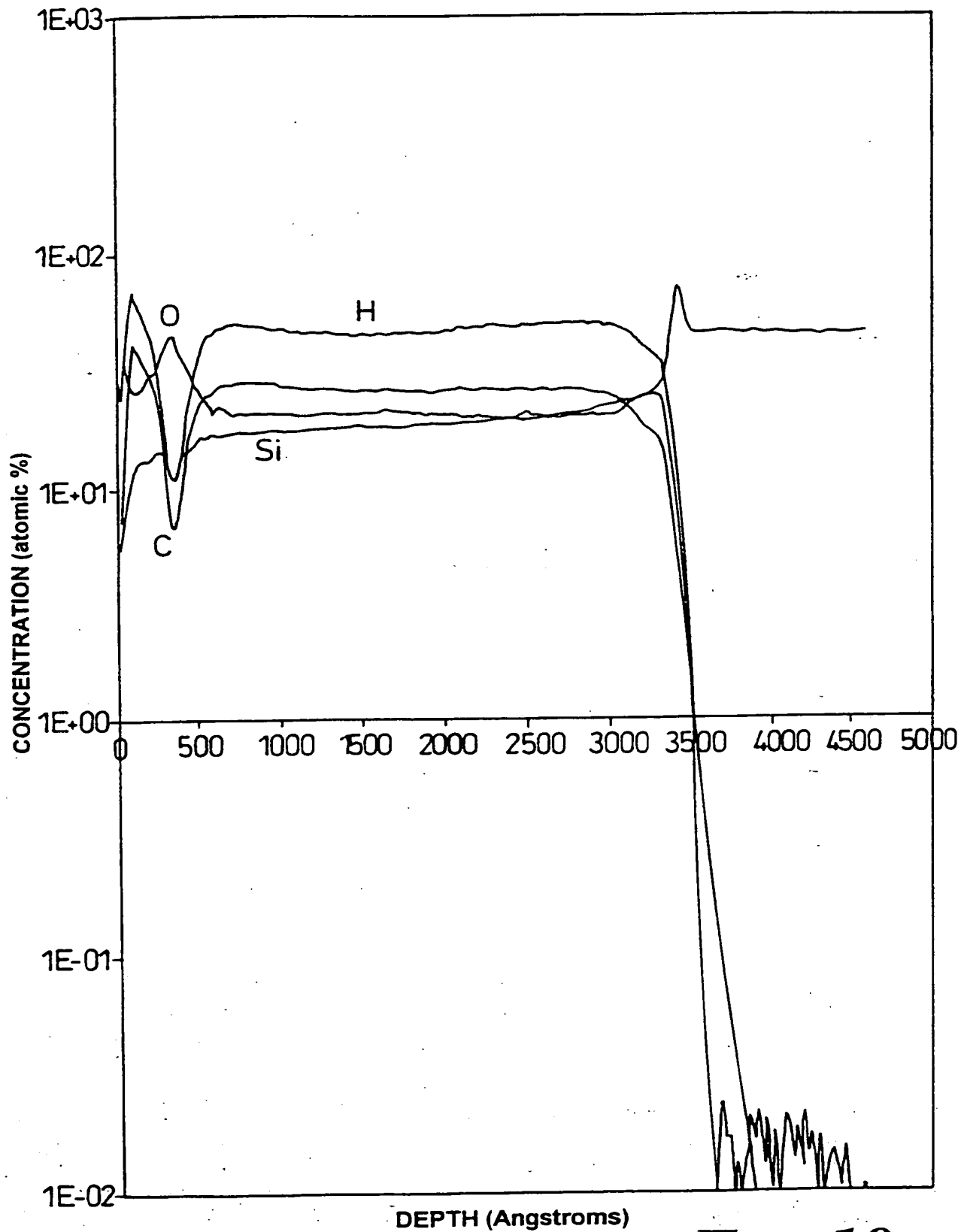


Fig. 18

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*Fig 19*

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PCT/GB 00/02301

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 00/02301

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